Date: July 18, 2000

Subj: Laboratory Audit at Research Triangle Institute (RTI)

From: Michael Clark, Team Leader, NAREL
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To: James Homolya, OAR, OAQPS

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On December 14, 1999, Michael Clark and Mary Wisdom, of the EPA's National Air and Radiation Environmental Laboratory (NAREL), with Dick Siscanaw, EPA-NE, conducted a systems audit of the Research Triangle Institute (RTI) facilities. The audit was conducted as part of the initial validation of RTI's analytical laboratory performance for the $PM_{2.5}$ Chemical Speciation Network.

At the initial briefing, Dr. Jayanty and members of the RTI staff gave the audit team an overview of RTI operations, coordination with OAQPS, and project-specific processes. RTI staff then conducted a tour for the audit team, during which the team met analysts, viewed equipment and facilities, and inspected documentation. Unfortunately the $PM_{2.5}$ Network was not operating at the time of the audit and field samples were not being analyzed by RTI. The audit was conducted looking at the systems RTI has put in place preparing to receive field samples.

Dick Siscanaw, EPA-NE, audited the analytical systems for total carbon analyses; his report is included as an attachment to this report and findings and recommendations for total carbon analyses are included in that report. The following are additional recommendations RTI should consider implementing.

Recommendations: All entries in logbooks should be signed and dated by the person making the entry. Signature and date should immediately follow the entry, on the same page.

All manual data entry should be proofed and verified by a second person. If possible, a system of double-entry for verification could be initiated so that verification must be done before entries are accepted into the system for further processing.

A regular system of calibration checks should be made of all balances, using Class I weights. The results of the checks should be documented in a balance logbook. Weights for the calibration checks should span the range of weights of expected samples. Criteria for a balance being out of calibration should be established, and procedures for taking a balance out of service and corrective action should be created.

When samples are received at RTI and problems are found with preservation, shipping, documentation, or other, immediate feedback to the field sampling team would enable the team to adjust its procedures before additional samples are taken with the same errors.

Although Ependorff-type pipets are factory calibrated, it is good practice to run a series of calibration checks each time the pipets are used. Dispensed aliquots of distilled water are weighed and compared to the nominal value of the pipet. Pipet calibration checks should be documented in a logbook. Criteria for failure, and processes for taking a pipet out of use and corrective action should be created.

For most analytical instruments, it is recommended that a daily calibration check sample be included at the end of each analytical run. This will ensure that the instrument is still in calibration after running all samples.

In refrigerator areas where samples are stored, a calibrated thermometer should be placed in a glycerol solution. Temperatures should be taken and recorded each day. Criteria for unacceptable temperature deviations and corrective action should be created.

The audit team appreciates the cooperation of the RTI staff during this audit. The RTI staff are professional and competent. Generally, we found the systems to be well thought out and implemented, and found no major problems or violations of good laboratory practices. It is clear that logistics for sample handling, documentation processes, and sample identification and coding systems have been well designed and are consistently followed. As part of the $PM_{2.5}$ Chemical Speciation QA Program NAREL plans to conduct a second audit of the RTI laboratory during FY00 while the $PM_{2.5}$ network is operating.

attachments: Audit Report from Dick Siscanaw, EPA-NE

Attachment: Audit Report on Carbon Analysis

Date: January 13, 2000

Subj: Laboratory Audit at Research Triangle Institute (RTI), 3040 Cornwallis Rd, RTP, NC

From: Dick Siscanaw, Chemist, EPA-NE

Thru: Rob Maxfield, Branch Chief, EPA-NE

To: Michael Clark, Team Leader, NAREL

File: 000100008.wpd

Introduction:

The laboratory audit was done on December 14, 1999 for the total carbon analyses on quartz fiber filters; this includes the measurements for total carbon (TC), organic carbon (OC), elemental carbon (EC) and carbonate carbon (CC). Unfortunately, the PM 2.5 mini trend program was not completed at the time of the audit and there were no field samples to review. This evaluation was based on the total carbon analyses for the Atlanta study. The equipment, data, and record books were evaluated for adherence to RTI's Standard Operation Procedure for the Determination of Organic, Elemental, Carbonate, and Total Carbon in Particulate Matter using a Thermal/Optical Carbon Analyzer, RTI's Quality Assurance Project Plan (QAPP), Chemical Speciation of PM2.5 Filter Samples and the NIOSH method 5040, Elemental Carbon (Diesel Particulate). The NIOSH method is the reference for the PM 2.5 program.

Personnel Interviewed:

Dr. M.(Max) R. Peterson
Melville Richards
Jim O'Rouke

Equipment:

Sunset TOT Instrument,
Mettler AT 400 Analytical Balance
Lindberg/Blue M Box Furnace

Results:

1- Finding: The weekly initial calibration was not done for samples analyzed on 9/22 - 28/99. The initial calibration was done on 9/14/99. The next calibration was done on 10/12/1999.

Recommendation: A full calibration must be performed at least weekly, per RTI's SOP, section 9.2.2. The weekly methane calibration should be done at the beginning of the week before any samples are analyzed. The least-squares correlation coefficient (r^2) criteria in the SOP is 0.98. I recommend reviewing your data after accumulating ten data points to determine if a 0.99 criteria is possible, RCRA method 8000B. The data reviewed was 0.999 (excellent).

2- Finding: No daily check standard was analyzed during 9/24 - 27/99. A daily check standard was analyzed on 10/7/99.

Recommendation: A daily check standard should be analyzed in the beginning of the sample batch before any samples are analyzed, per RTI's SOP, section 9.2.1. I also recommend running a check standard at the end of the sample batch to bracket the samples. The final check standard is used to verify that the instrument was within calibration for the entire sample batch.

3. Finding Only one duplicate was included with the 20 samples analyzed on 10/7/99. All of the remaining analyses reviewed had at least a 10% duplicate frequency. The laboratory has not established acceptance criteria for the duplicate data.

Recommendation: Duplicates must be performed at a 10% frequency, per RTI's SOP, Section 9.3. These values must be evaluated and recorded in a quality control table or chart. RTI will need to establish QC windows as they accumulate data. I recommend using EPA criteria to start with.

Concentrations (ug/cm^2)	Acceptance Criteria
Values greater than 10	Less than 10% RPD
5 - 10	Less than 15% RPD
Values less than 5	Within 0.5 ug/cm^2

4. Finding: No thermometer in the sample storage freezer.

Recommendation: All sample and standards refrigerators/freezers should be monitored

daily. The typical acceptance range for a freezer is -10 to -20 C.

5- Finding — The preparation of the sucrose standard on 9/24/99 was not recorded in the reagent preparation log book. A previously prepared standard was recorded. The balance was calibrated on 8/5/99.

Recommendations: All standards must be recorded in the reagent preparation log book, per RTI's QAPP, table A.9.4. I also recommend checking the balance before the standard is prepared with one or two class 1 weights. These check weights should be traceable to a certified NIST reference and the check data recorded in a log book which is kept at the balance. This is a common practice throughout the Northeast environmental and health labs. It is also being proposed by National Environmental Laboratory Accreditation Conference (NELAP), balance check calibration, for accreditation.

6. Finding: The RTI acceptance criteria for the laboratory instrument blank is high, 1 ug/cm^2. All the observed values were below 0.3 ug/cm^2.

Recommendation: Collect 25 instrument blank values and then establish a statistical criteria that is more reasonable or use 0.3 ug/cm^2. Both, EPA and DRI use 0.3 ug/cm^2 as a limit for their instrument blanks.

7. Finding: RTI has not established a holding time for their pre cleaned filters. Since no samples were available at the time of the audit, this point could not be investigated. A precleaned quartz filter will start absorbing background organic compounds as soon as it is cleaned. The acceptance criteria for the precleaned filters, 1 ug/cm^2 , is probably high.

Recommendation: RTI should verify that precleaned filters will meet the established background levels during the anticipated holding time period prior to shipment to the field. Once established, the holding time should be added to RTI's SOP, section 4.5 and RTI's SHAL SOP, section 23, step 5. Also, data should be accumulated for the observed values in a database and used to establish a true statistical limit for these operation.

8-Finding — The temperature set points and residence times on the thermogram appear different from the NIOSH 5040 method. The thermogram is the temperature profile of the total carbon analysis.

Recommendations: It appears that the instrument has had some maintenance performed

between 10/5 and 10/15/99 and there was a visible change in the operating conditions. Strangely, the older thermograms appear to be more consistent with the NIOSH 5040 method, i.e. more of a plateau at the set points. The actual temperature and residence time at each set point should be measured and recorded in the maintenance book. Presently, matching the NIOSH 5040 is not critical for the total carbon determination, however this may become an issue if fractionation of the OC and EC is required in the future. The data for the split samples analyzed by RTI compared favorably with data generated by EPA, NIOSH, DRI, and Sunset laboratory.

9. Finding: RTI has never performed the carbonate digestion procedure.

Recommendation: This is in RTI's SOP, section 7.4.2. The laboratory should run some blanks, standards and samples to confirm this step in their SOP. The SOP is confusing because RTI uses this procedure to confirm the carbonate value manually integrated in step 7.4.1. Clarification would be appropriate.

10. Finding: No recent data was available for the calibration of the mass flow controllers (MFCs) used in the instrument or for the determination of the transit time. The transit time is the time it takes for the carbon dioxide produced from the carbon in the sample and pass through the methanator to the flame ionization detector. This is critical information needed to establish the split point between the OC and EC fractions. RTI's transit time is 6 sec which is lower than normal.

Recommendations: These checks were performed during the instrument's installation which was several years ago. The MFCs should be checked and recorded in the maintenance book annually or with the replacement of the methanator. The transit time of 6 sec should be confirmed with the parameter file, transit.par.

Conclusion:

I want to thank Max and Melville for their time and cooperation. They are qualified, highly competent and conscientious about doing a fine job. I enjoyed spending the time and sharing information with them. There are many positive actions RTI has taken to assure the quality of the total carbon analyses they will be performing, for example, the data for their method detection level study was 0.124 ug/cm^2 (NIOSH is 0.15 ug/cm^2); monitor the response factors to check their methanator; variance of the daily methane counts was only 1% RSD, meaning the instrument is extremely stable and reproducible. Overall, RTI has no major problems performing the total carbon analyses for the PM 2.5 program.